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Subject INTERIM RESEARCH REPORT

FOR

RADIOPHOTOLUMINESCENT AND TENEBRESCENT GLASSES

NAVY DEPARTMENT BUREAU OF SHIPS ELECTRONICS DIVISIONS

NObsr-57016 NE-051551 2.3

By N . J. Kreidl

Chemical Research Director

Bausch & Lomb Optical Co.

Rochester 2, New York

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BAUSCH & LOMB OPTICAL CO.

ROCHESTER 2, NEW YORK

June 24, 1953

Office of the Chief Signal Officer
Engineering and Technical Division
Washington 25, D. C.
Att: SIGGG-P

Subject: Contract NObsr-57016
Interim Research Report

Gentlemen:

Enclosed are your copies of the following Interim Research Report in connection with the subject contract:

Interim Research Report
Contract NObsr-57016
for the period 24 September
to 24 December 1952

Very truly yours,

BAUSCH & LOMB OPTICAL CO.

C Wolfard

Defense Contract Department

CTWolfard/sm
Encs.

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1853 1953 INTO A NEW CENTURY OF OPTICAL PROGRESS

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INTERIM RESEARCH REPORT

FOR

RADIOPHOTOLUMINESCENT AND TENEBRESCENT GLASSES

This report covers the period 24 September 1952 to 24 December 1952.

BAUSCH AND LOMB OPTICAL CO.

ROCHESTER 2, NEW YORK

NAVY DEPARTMENT BUREAU OF SHIPS ELECTRONICS DIVISIONS

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1. PART I

PRESENT STATUS

1.1 PURPOSE

The purpose of this investigation is to determine, in glasses, the causes of optical changes produced by X- or gamma radiation, to select and develop compositions which combine optimum indication and integration of X- or gamma radiation with desirable technological properties affecting their eventual manufacture, and to evaluate glasses which without radiation by X- or gamma rays will fluoresce in a manner similar to the existing dosimeter glass.

1.2 GENERAL FACTUAL DATA

1.2.1 Personnel

	Hours Spent by Personnel on this Project		
Month Ending	24 Oct.	23 Nov.	27 Dec.
N. J. Kreidl Director of Research	31	36	-
T.G. Pett Head, Glass Physics Section	15	5	15
G. Blair Chemist	160	160	160
W. Kirchgessner Spectroscopist	32	9-1/2	21
J. Mogenhan Chemist	8	8	9
W. VandeMark Technician	13	8	-
G. Biel Technician	-	20	12
E. Herko Chemist	-	2	-
E. Simms Technician	-	1	-

1.2.2 Test Equipment

CP-95 (XN-3)/PD Radiac Computer Indicator, Serial No. 19 together with calibration standards supplied by the Polaroid Corporation, was used to obtain all readings on the radiophotoluminescent glass. Readings are given in N.U. (New Units). This unit was established by Dr. J. Schulman to relate a gamma radiation dose of 100r with a 100 unit reading with this instrument on a glass of specified sensitivity. In other words under proper conditions, N.U. readings will indicate the dosage in roentgens.

1.2.2 Test Equipment (Continued)

A Co^{60} source was used for all exposures. This equipment was described in Interim Report 24 Feb. to 24 March 1952.

A Hunter Color-Color Difference Meter was used in evaluating color changes. This instrument is essentially a photometer measuring three factors - transmittance and 2 chromaticity factors. (Reference: R. S. Hunter, "Photoelectric Color-Color Difference Meter", J. Opt. Soc. Am., 38, (7), 661 (1948).

A Hardy Recording Spectrophotometer was used in evaluating changes of absorption in the visible range of the spectrum.

1.3 DETAIL FACTUAL DATA

1.3.1 Topic 1 - Present Radiophotoluminescent Glass

1.3.1.1 Sensitivity and Predose

1.3.1.1.1 Purpose

To classify available raw materials in relation to the present specifications on predose and sensitivity of the radiophotoluminescent glass specified for use in Radiac Detector DT-60/PD, Specification - MIL-D-16153C (Ships).

1.3.1.1.2 Experimental

As the result of discussions and correspondence with the Victor Chemical Works, it was suspected that iron might have been introduced into the rather abrasive aluminum metaphosphate from the iron equipment used in the milling operation. New raw materials supplied by Victor were used in making melts and the glasses were tested.

1.3.1.1.3 Results and Conclusions (Table I)

The results, as seen in Table I in comparing melts 1A4089 and 1A4105, verified our suspicions. The sensitivity of the glass increased from 43 to 128 N.U. when unmilled was substituted for milled material. Unmilled aluminum metaphosphate did not blend well with other batch materials, but satisfactory results were also achieved with aluminum metaphosphate that was milled in non-metallic equipment (Lot #E-9318), (Melt #1A4476). Crushed aluminum metaphosphate (Lot #E-8838), (Melt #1A4297) was found to be inferior.

TABLE I

Dosimeter Sensitivity and Predose as a Function of Batch Variation

Melt No.		Batch		Remarks	
1A4089	$Al(PO_3)_3$	Victor - Lot #E-8768 (Milled in iron equipment)	(50%)	Readings:	N.U.
	$Ba(PO_3)_2$	Monsanto - Lot #20607	(25%)		
	KPO_3	Monsanto - Lot #2	(20%)	Predose	23
	KNO_3	Plant Material	(5%)	Sensitivity	43
	$AgPO_3$	Monsanto - Lot #70293	(8%)		
1A4105	Same as 1A4089 except with the substitution of:				N.U.
	$Al(PO_3)_3$	Victor - Lot #E-8760 (Unmilled)	(50%)	Predose	24
				Sensitivity	128
1A4297	$Al(PO_3)_3$	Victor - Lot #E-8838 (Crushed)	(50%)		N.U.
	$Ba(PO_3)_2$	Monsanto - Lot #50020	(25%)		
	KPO_3	Victor - Lot #E-9082	(20%)	Predose	44
	KNO_3	Plant Material	(5%)	Sensitivity	81
	$AgPO_3$	Monsanto - Lot #70293	(8%)		
1A4476	Same as 1A4297 except with the substitution of:				N.U.
	$Al(PO_3)_3$	Victor - Lot #E-9318 (Milled in non-metallic equipment)	(50%)	Predose	36
				Sensitivity	114

1.3.2 Topic 2 - Standard Glass

1.3.2.1 Purpose

At the request of Mr. Rainey, Bureau of Ships, work was again resumed on this phase of the program to investigate glasses that might have an intensity of fluorescence similar to a silver phosphate glass dosed to 150r in respect to performance and possible improvements of durability.

1.3.2.2 Experimental

Experiments on performance of the present E-9272 type were started. Two experimental melts were made, that were slight revisions of a composition (Melt No. 1A4133) previcusly tried for the Polaroid Corporation, that had given very satisfactory performance, in the hope of improving its durability (see letter of 24 June 1952 to Chief, Bureau of Ships, Code 854, from Dr. W. A. Shurcliff).

1.3.2.3 Results and Conclusions (Table II)

Results on the performance of the present E-9272 will be discussed in the next report as the work is still in progress.

Results on durability improvements were considered unpromising as the chemical changes considered necessary adversely affected fluorescence performance and further work was discontinued in agreement with the Bureau of Ships.

TABLE II

Attempts to Improve the Durability of the Manganese Standard Glass

	Composition - Wt.%		
Oxide	Melt No. 1A4395	Melt No. 1A4497	
SiO_2	41.5	37.41	*
Na_2O	23.0	26.06	*
B_2O_3	32.5	36.51	*
MnO_2	0.01	0.01	*
As_2O_3	0.0005	0.0005	*
Sb_2O_3	0.0005	0.0005	*
MgO		1.0	**
CaO		2.0	**
ZrO_2		1.0	**
Al_2O_3	3.0	2.0	**
Color of Fluorescent Light	Yellow	Orange	
Intensity	Approx. 150 N.U.	Approx. 150 N.U.	
Durability	No Improvement	No Improvement	

* This is composition of Melt No. 1A4133, see text.

** Added in excess of 100% to this base.

1.3.3 Topic 3 - Tenebrescent Glass

1.3.3.1 Purpose

Investigation of causes of changes in absorption under irradiation, especially if useful in the development of glasses for direct reading dosimeters ("tenebrescent glasses").

1.3.3.2 Experimental

An experimental remelt (1A4248) was made in a 4" zircon crucible and 2 blocks of glass were cast of the most promising composition previously reported, viz.:

$Al(PO_3)_3$	-	50 wt.%
$Ba(PO_3)_2$	-	25 wt.%
KPO_3	-	20 wt.%
KNO_3	-	5 wt.%
$AgPO_3$	-	.8 wt.% } Added in excess of 100%
BaF_2	-	1 wt.% }

The blocks were finished in the following manner:

Dimensions - 39 x 48.5 x 57mm thick.

Finish - The 2 (39 x 48.5) surfaces were polished.

The other 4 surfaces were ground to #80 grit finish and painted with black paint.

Readings were taken of the absorption bands formed in the visible spectrum after exposures of 20, 40, 80, 160, 320, 640r on a Hardy Recording Spectrophotometer and a Hunter

Color-Color Difference Meter (see Figs. 1, 2, 3).

1.3.3.3 Results and Conclusions

Both the Hardy and Hunter instruments give measurable and linear readings starting at 20r exposure as can be seen from Figs. 1, 2, 3. A visible change was detected on the exposed sample at the 80r level. A control sample was used on all readings. All these findings are rather unique at the present stage of knowledge. Possibly the color change could be enhanced by using greater thickness and by the use of an integrating device such as coating a long cylinder with white paint and enclosing this assembly in an opaque container so that only one surface would be exposed, as suggested by Dr. J. Schulman of the Naval Research Laboratory. Also, the strong absorption band formed by irradiation in the near U.V. may permit the use of the emission change in a fluorescent material to obtain a more sensitive response.

The direct coloration of glass may be useful in a direct reading dosimeter if greater sensitivity can be obtained. However, this glass is not now considered practical, as an untrained observer will not easily recognize a change much below 200r in a piece of the largest practical dimensions, since it happens to occur in a part of the spectrum to which the human eye is most insensitive.

2. PART II

PROGRAM FOR NEXT INTERVAL

2.1 In agreement with Mr. Rainey, Bureau of Ships, the remaining time on this contract will be utilized to complete work on three phases, namely:

a) The investigation of the effect of heat treatment to erase previous exposure history in the radiophotoluminescent dosimeter glass and to evaluate second exposure sensitivity after this erasing.

b) The investigation of reproducibility of the manganese standard glass (E-9272 type) in production melting.

c) Whatever may be found necessary to complete the contract adequately.

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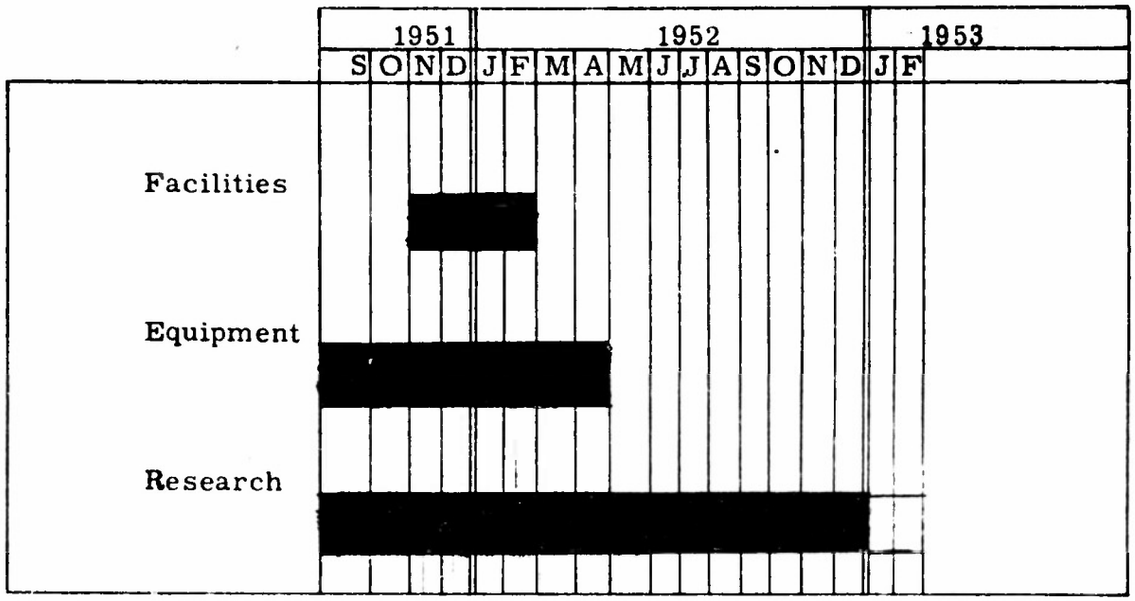
Project Performance and Schedule
Index No. NE-051551

Contract NObsr-57016 Report Date 24 December 1952

Period Covered 24 September 1952 to December 1952



Projected Work

Completed Work

Fig. - 1

Sensitivity of a Direct

Reading Dosimeter Glass

by Colorimetry.

Melt No. 1A4248 - Two samples.
Sample dimensions - 39mm X 48.5mm X 57mm thick.
Sample No. 1 used as control in taking all readings.
Sample No. 2 exposed to 20, 40, 80, 160, 320, 640 r.
Readings were taken on a Hunter Color - Difference Meter after each exposure.

By the use of the Formula - I (Color - Difference) = a + b + (0.5 x R) The readings were converted to Judd Units.

A Judd is defined as the least perceptible change in color that is commercially practical

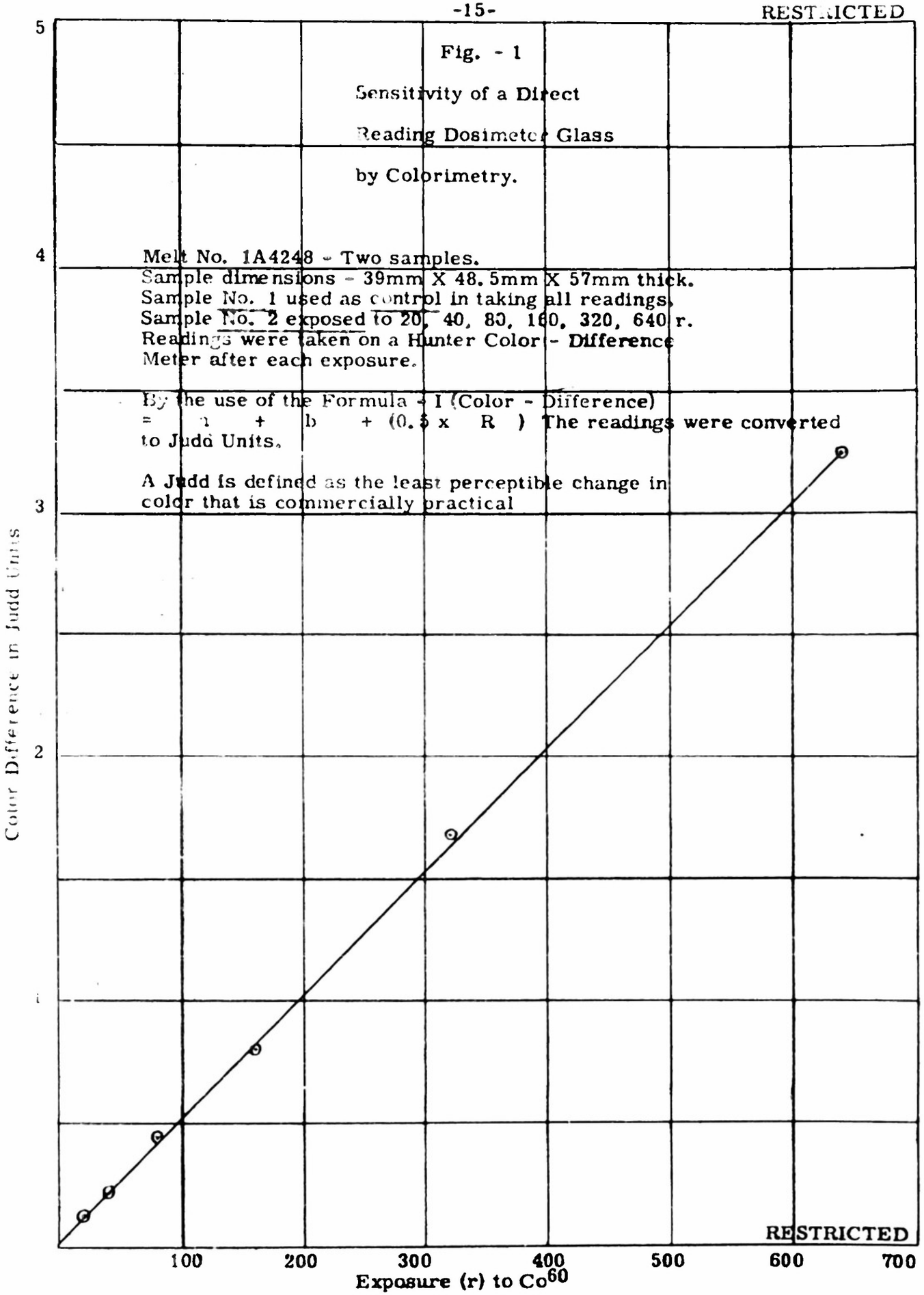
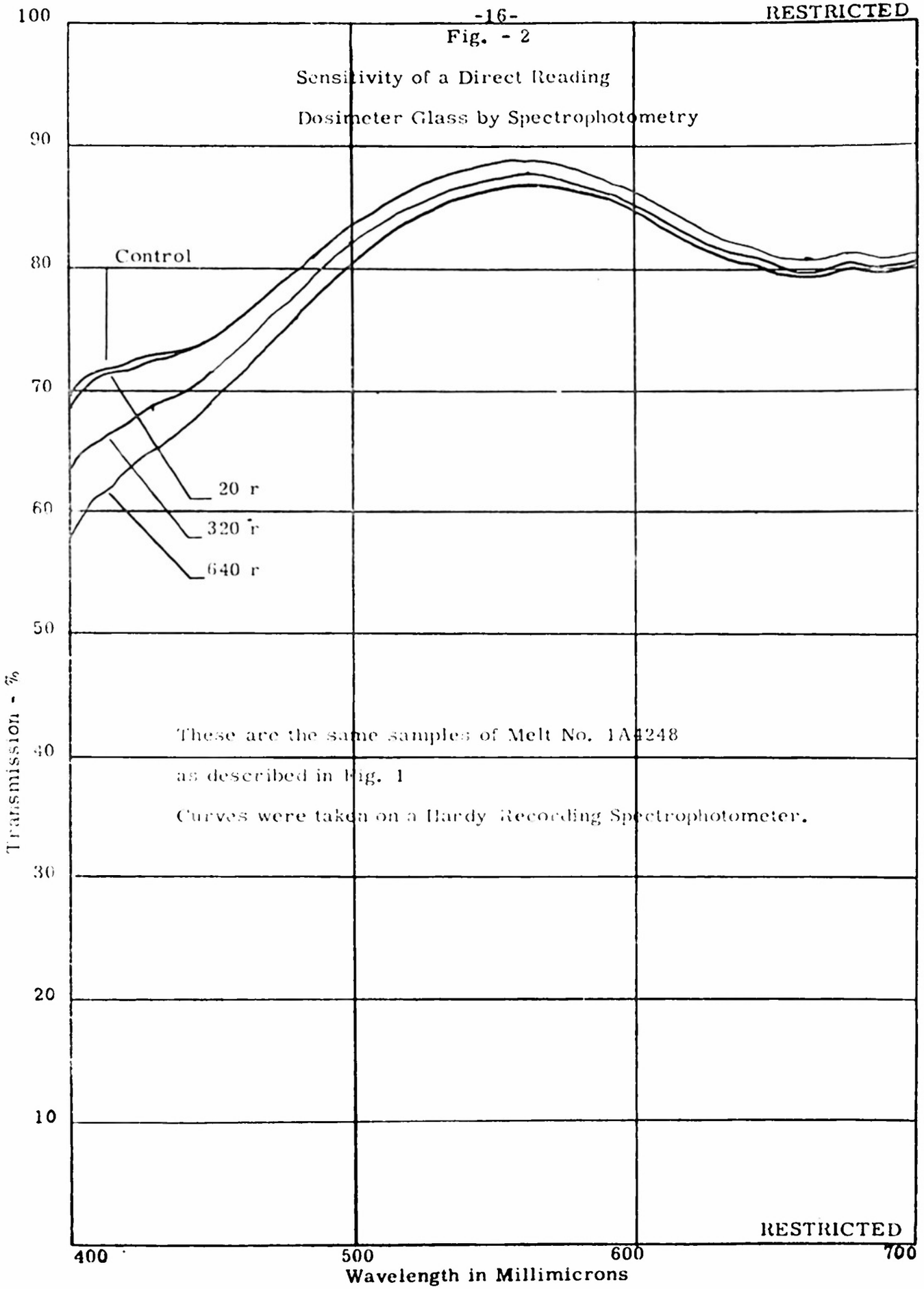


Fig. - 2

Sensitivity of a Direct Reading

Dosimeter Glass by Spectrophotometry



These are the same samples of Melt No. 1A4248 as described in Fig. 1

Curves were taken on a Hardy Recording Spectrophotometer.

Fig. - 3

Sensitivity of a Direct Reading Dosimeter Glass by Density Difference calculated from Spectrophotometric curves (Fig. 2)

These are the same samples of Melt No. 1A4248 as in Fig. 1